ecology and environment, inc.
101 YESLER WAY, SEATTLE, WASHINGTON, 98104, TEL. 206/624-9537
International Specialists in the Environment

May 10, 1988

Jeff Webb
Environmental Protection Agency
1200 Sixth Avenue, HW-113
Seattle, WA 98101

Ref: TDD T10-8707-009

Dear Jeff:

Enclosed is a revised version of the QA Memo for dioxin analyses of two samples from the Corigliano site in Spokane, WA. Discussions between the chemist and the analytical laboratory have satisfied the chemist that no dioxin/furan homologues were found in either sample. This is reflected in the revisions to sections XI and XII.

Sincerely,

Priscilla Anderson

Priscilla Anderson
Toxicologist

PNA/jcw

Enclosure

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MEMORANDUM

DATE: March 22, 1988

TO: Priscilla Anderson, TATM-Toxicologist, E&E, Seattle

FROM: Roger McGinnis, Chemist, E&E, Seattle RM

SUBJ: **Polychlorinated dibenzodioxin/furan Homologue Analysis Data Quality Assurance Review for Corigliano Landfill**

REF: TDD: T10-8707-009
PAN: TWA-0526-RFA

The quality assurance review of two soil samples collected from Corigliano Landfill has been completed. The samples were analyzed for polychlorinated dibenzodioxins/furans (PCDD/PCDF) by TMS Analytical Services of Indianapolis, Indiana.

The samples were numbered:

T7080035 and T7080036.

Data Qualifications

Samples were analyzed by Tandem Mass spectroscopy (MS/MS). While no USEPA protocols have been established for this method data review was performed using modified USEPA Method 8280 criteria.

I Timeliness

No holding time criteria have been established. Sample T7080036 was extracted and analyzed on the day received. Sample T7080035 was analyzed 24 days after receipt.

II PCDD/PCDF Analysis

The laboratory did not specify the chromatographic column used for separation. However, they stated a short, non-polar column was used rather than the standard, longer, polar column.

III Initial Calibration

a) Sensitivity

The signal/noise ratio for internal standards was greater than 10.

b) Relative Response Factors

No criteria have been established for internal standard RRFs. The data summary sheet was missing for run 1 of standard solution 1.

c) Linearity

Internal standard RRFs met the laboratory criteria of less than 10% relative standard devition (RSD).

d) Ion Ratios

Laboratory criteria for ion ratios were met. Ratios were independent of concentration with less than 10% RSD.

e) Surrogate Standards

No surrogate raw data including areas, ion ratios or relative response factors were supplied by the laboratory.

IV Continuing Calibration

a) Relative Response Factors

Percent difference between initial calibration and continuing calibration RRFs exceeded 10% for a number of dioxin/furan congeners. Tabulated areas for HpCDD/HpCDF do not match raw data for sample T7080035.

Date	Congener	% Difference	QA Limit
8/28/87	TCDD	12.3%	10%
8/28/87	TCDF	24.0%	10%
8/28/87	PCDD	11.0%	10%
8/28/87	PCDF	21.6%	10%
8/28/87	HPCDF	10.5%	10%
8/28/87	OCDF	12.4%	10%
9/21/87	TCDF	14.7%	10%
9/21/87	PCDF	21.6%	10%

The laboratory did not perform a new initial calibration as required by their quality control protocols.

b) Ion Ratios

The percent difference between initial and continuing calibration ion ratios was 14.4% for OCDD. The laboratory did not perform a new initial calibration.

V Laboratory Method Blank

No chlorinated dioxins or furans were detected in the laboratory blank. The laboratory stated ion ratios did not meet criteria for

positive identification though no raw data were provided.

VI Duplicate Sample Analysis

No duplicate analysis was performed.

VII Native Spike Analysis

No native spike analysis was performed by the laboratory.

VIII Field Blank Analysis

No field blank was submitted to the laboratory.

IX Performance Evaluation Sample

No performance evaluation sample was submitted to the laboratory.

X Surrogate Analysis

Reported Surrogate recoveries for several samples were outside laboratory quality control limits. No data were present to verify results.

Sample	Surrogate	% Recovery	QA Limits
Method Blank	Hepta/Octa	31.88	60-140%
T7080035	Hexa/Hepta	54.01	60-140%
T7080035	Hepta/Octa	38.38	60-140%
T7080036	Tetra/penta	196.27	60-140%

XI Sample Analysis

Ion chromatograms of sample T7080035 and T7080036 showed peaks in dioxin/furan windows. The laboratory stated ion ratios did not meet matching criteria though no data were present to verify ratios.

XII Data Assessment

Data copy quality was unacceptable. Many raw data sheets were illegible and poorly labeled.

The laboratory was contacted on May 4, 1988 to clarify detection limit calculations.

Sample quantitation limits are considered estimated quantities only (flagged J) since no data are present regarding duplicate, native spike, and performance evaluation samples. In addition, the continuing calibration did not meet criteria.

dioxin
T7080035, 36

TMS
TANDEM MASS SPECTROMETRY

TMS ANALYTICAL SERVICES, INC.

6376 Morenci Trail
Indianapolis, Indiana 46268
317-291-5697

rec'd 10/23

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GC/MS/MS ANALYSIS REPORT FORM
ANALYSIS FOR POLYCHLORINATED DIOXINS AND FURANS

CLIENT: ECOLOGY & ENVIRONMENT
SAMPLE: METHOD BLANK T-SERIES

ANALYTE	RESULT TOTAL PCDD/PCDF'S ng/g	ESTIMATED LIMIT OF DETECTION ng/g	TCDD RESULT 2,3,7,8-WINDOW ** ng/g	TCDD ESTIMATED LIMIT OF DETECTION 2,3,7,8-WINDOW ** ng/g
TCDD = TETRACHLORODIBENZODIOXIN	NONE DETECTED	0.04 J	NONE DETECTED	0.04 J
TCDF = TETRACHLORODIBENZOFURAN	NONE DETECTED	0.03		
PCDD = PENTACHLORODIBENZODIOXIN	NONE DETECTED	0.08		
PCDF = PENTACHLORODIBENZOFURAN	NONE DETECTED	0.05		
HXCDD = HEXACHLORODIBENZODIOXIN	NONE DETECTED	0.26		
HXCDF = HEXACHLORODIBENZOFURAN	NONE DETECTED	0.27		
HPCDD = HEPTACHLORODIBENZODIOXIN	NONE DETECTED	0.29		
HPCDF = HEPTACHLORODIBENZOFURAN	NONE DETECTED	0.16		
OCDD = OCTACHLORODIBENZODIOXIN	NONE DETECTED	1.26		
OCDF = OCTACHLORODIBENZOFURAN	NONE DETECTED	1.66		

ng/g = parts-per-billion
ng/kg = parts-per-trillion

THIS IS THE METHOD BLANK WHICH WAS RUN ALONG WITH THIS SAMPLE SET; THE METHOD BLANK SHOWED NONE-DETECTED RESULTS FOR ALL ANALYTES

LOD = LIMIT OF DETECTION CALCULATED AS EQUIVALENT OF 2.5 X PEAK-TO-VALLEY NOISE PEAK HEIGHT OF 2ND DAUGHTER ION COMPARED TO PEAK HEIGHT OF INTERNAL STANDARD.

NOTE: FOR ALL POSITIVE RESULTS THE ION RATIOS FOR DAUGHTER IONS WERE ALL WITHIN 15% OF CALIBRATION AVERAGES

** NOTE: THE 2,3,7,8-TCDD WINDOW IS ONLY SEMI-ISOMER SPECIFIC IN THAT A SHORT, NON-POLAR CAPILLARY COLUMN WAS USED FOR SCREENING PURPOSES. THIS COLUMN SEPARATES THE THE 2,3,7,8-TCDD ISOMER FROM ALL OTHER ISOMERS EXCEPT THE TWO CLOSEST ELUTING ISOMERS. ACCORDINGLY, THIS NUMBER SHOULD BE REGARDED AS THE MAXIMUM 2,3,7,8-TCDD TO BE EXPECTED UPON ISOMER SPECIFIC ANALYSIS. TO PROPERLY USE THIS RESULT, IT IS ESSENTIAL TO UNDERSTAND THAT UPON ANALYSIS USING A LONGER POLAR CAPILLARY COLUMN MEETING EPA CRITERIA FOR ISOMER SPECIFICITY, THAT ANY VALUE, INCLUDING ZERO, BELOW THIS VALUE MAY BE OBTAINED. SUCH WIDE DIFFERENCES ARE RELATIVELY RARE, AND SINCE THE ASSUMPTIONS ERR ON THE SIDE OF SAFETY THE VALUES ARE USEFUL FOR SCREENING PURPOSES. NEGATIVE, OR NONE-DETECTED RESULTS ARE DEFINITIVE AND NO FURTHER WORK NEED BE DONE EVEN IF ISOMER SPECIFICITY IS DESIRED.

TMS
TANDEM MASS SPECTROMETRY

TMS ANALYTICAL SERVICES, INC.

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Indianapolis, Indiana 46268
317-291-5697

GC/MS/MS ANALYSIS REPORT FORM
ANALYSIS FOR POLYCHLORINATED DIOXINS AND FURANS

CLIENT: ECOLOGY & ENVIRONMENT
SAMPLE: T7080035

ANALYTE	RESULT TOTAL PCDD/PCDF'S ng/g	ESTIMATED LIMIT OF DETECTION ng/g	TCDD RESULT 2,3,7,8-WINDOW ** ng/g	TCDD ESTIMATED LIMIT OF DETECTION 2,3,7,8-WINDOW ** ng/g
TCDD = TETRACHLORODIBENZODIOXIN	NONE DETECTED	0.23 J	NONE DETECTED	0.23 J
TCDF = TETRACHLORODIBENZOFURAN	NONE DETECTED	0.21		
PCDD = PENTACHLORODIBENZODIOXIN	NONE DETECTED	0.20		
PCDF = PENTACHLORODIBENZOFURAN	NONE DETECTED	0.18		
HXCDD = HEXACHLORODIBENZODIOXIN	NONE DETECTED	0.76		
HXCDF = HEXACHLORODIBENZOFURAN	NONE DETECTED	0.51		
HPCDD = HEPTACHLORODIBENZODIOXIN	NONE DETECTED	1.56		
HPCDF = HEPTACHLORODIBENZOFURAN	NONE DETECTED	1.09		
OCDD = OCTACHLORODIBENZODIOXIN	NONE DETECTED	4.36		
OCDF = OCTACHLORODIBENZOFURAN	NONE DETECTED	3.60		

ng/g = parts-per-billion
ng/kg = parts-per-trillion

AN ACCEPTABLE METHOD BLANK WAS RUN ALONG WITH THIS SAMPLE; THE METHOD BLANK SHOWED NONE-DETECTED RESULTS FOR ALL ANALYTES

LOD = LIMIT OF DETECTION CALCULATED AS EQUIVALENT OF 2.5 X PEAK-TO-VALLEY NOISE PEAK HEIGHT OF 2ND DAUGHTER ION COMPARED TO PEAK HEIGHT OF INTERNAL STANDARD.

NOTE: FOR ALL POSITIVE RESULTS THE ION RATIOS FOR DAUGHTER IONS WERE ALL WITHIN 15% OF CALIBRATION AVERAGES

** NOTE: THE 2,3,7,8-TCDD WINDOW IS ONLY SEMI-ISOMER SPECIFIC IN THAT A SHORT, NON-POLAR CAPILLARY COLUMN WAS USED FOR SCREENING PURPOSES. THIS COLUMN SEPARATES THE THE 2,3,7,8-TCDD ISOMER FROM ALL OTHER ISOMERS EXCEPT THE TWO CLOSEST ELUTING ISOMERS. ACCORDINGLY, THIS NUMBER SHOULD BE REGARDED AS THE MAXIMUM 2,3,7,8-TCDD TO BE EXPECTED UPON ISOMER SPECIFIC ANALYSIS. TO PROPERLY USE THIS RESULT, IT IS ESSENTIAL TO UNDERSTAND THAT UPON ANALYSIS USING A LONGER POLAR CAPILLARY COLUMN MEETING EPA CRITERIA FOR ISOMER SPECIFICITY, THAT ANY VALUE, INCLUDING ZERO, BELOW THIS VALUE MAY BE OBTAINED. SUCH WIDE DIFFERENCES ARE RELATIVELY RARE, AND SINCE THE ASSUMPTIONS ERR ON THE SIDE OF SAFETY THE VALUES ARE USEFUL FOR SCREENING PURPOSES. NEGATIVE, OR NONE-DETECTED RESULTS ARE DEFINITIVE AND NO FURTHER WORK NEED BE DONE EVEN IF ISOMER SPECIFICITY IS DESIRED.

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GC/MS/MS ANALYSIS REPORT FORM
ANALYSIS FOR POLYCHLORINATED DIOXINS AND FURANS

CLIENT: ECOLOGY & ENVIRONMENT
SAMPLE: T7080036

ANALYTE	RESULT TOTAL PCDD/PCDF'S ng/g	ESTIMATED LIMIT OF DETECTION ng/g	TCDD RESULT 2,3,7,8-WINDOW ** ng/g	TCDD ESTIMATED LIMIT OF DETECTION 2,3,7,8-WINDOW ** ng/g
TCDD = TETRACHLORODIBENZODIOXIN	NONE DETECTED	0.38 J	NONE DETECTED	0.38 J
TCDF = TETRACHLORODIBENZOFURAN	NONE DETECTED	0.22		
PCDD = PENTACHLORODIBENZODIOXIN	NONE DETECTED	0.23		
PCDF = PENTACHLORODIBENZOFURAN	NONE DETECTED	0.26		
HXCDD = HEXACHLORODIBENZODIOXIN	NONE DETECTED	0.54		
HXCDF = HEXACHLORODIBENZOFURAN	NONE DETECTED	0.60		
HPCDD = HEPTACHLORODIBENZODIOXIN	NONE DETECTED	0.97		
HPCDF = HEPTACHLORODIBENZOFURAN	NONE DETECTED	1.13		
OCDD = OCTACHLORODIBENZODIOXIN	NONE DETECTED	1.78		
OCDF = OCTACHLORODIBENZOFURAN	NONE DETECTED	1.44		

ng/g = parts-per-billion
ng/kg = parts-per-trillion

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